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PATENT ABSTRACTS OF JAPAN

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(54) INTERMEDIATE FILM FOR SANDWICH GLASS AND SANDWICH GLASS

(57)Abstract:

PROBLEM TO BE SOLVED: To produce an intermediate film for sandwich glass which is excellent in the basic performance as the intermediate film for the sandwich glass, such as transparency, light resistance, weatherability, impact resistance and adhesiveness, has excellent moisture resistance and antistatic property, is free of the possibility of the outbreak of a fire during autoclaving in manufacturing the sandwich glass and is improved in trim cut workability for the ends of the sandwich glass.

SOLUTION: This intermediate film for the sandwich glass consists of 100 pts.wt. polyvinyl acetal resin, 20 to 60 pts.wt. triethylene glycol di-2- ethylhexanoate, 0.01 to 0.1 pt.wt. mixture composed of magnesium 2-ethylacetate and magnesium acetate and 0.005 to 0.5 pt.wt. 4-12C potassium carboxylate, in which the mixture satisfies the relation $0.5 < \text{magnesium 2-ethylacetate} / \text{magnesium acetate} < 3.0$ by weight.

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CLAIMS

[Claim(s)]

[Claim 1] The interlayer for glass laminates characterized by being the interlayer for glass laminates which the polyvinyl-acetal resin 100 weight section, TORIECHIRENGURIKORUJI 2-ethylhexanoate 20 - 60 weight sections, the mixture 0.01 of 2-ethyl butanoic acid magnesium and magnesium acetate - the 0.1 weight sections, and a carbon number become from the carboxylic-acid potassium salt 0.005 of 4-12 - the 0.5 weight sections, and being the mixture with which the above-mentioned mixture fills the relation between 0.5<2-ethyl butanoic acid magnesium / magnesium acetate <3.0 with a weight ratio.

[Claim 2] The interlayer for glass laminates according to claim 1 to which polyvinyl-acetal resin is characterized by being 66-72-mol the polyvinyl butyral resin it is [polyvinyl butyral resin] % by whenever [butyral-ized].

[Claim 3] The glass laminate characterized by making the interlayer for glass laminates according to claim 1 or 2 intervene between the glass of a pair, and making it come at least to unify.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the glass laminate which used the interlayer for glass laminates, and this interlayer.

[0002]

[Description of the Prior Art] Before, since it is rare for the fragment of glass to disperse and it is safe for the glass laminate with which it comes to fasten the interlayer for glass laminates between the glass plates of a pair even if it damages in response to an external impact, it is widely used for windowpanes, such as a vehicle like an automobile, an aircraft, and a building, etc. [at least] The interlayer which consists of polyvinyl-acetal resin plasticized with the plasticizer as an interlayer used for such a glass laminate has an adhesive property with outstanding glass, tough tensile strength, and high transparency, and is especially used suitably as a windowpane of vehicles, such as an automobile, etc. [0003] In recent years, the interlayer for glass laminates which the demand to the quality of an interlayer is severe, and adhesion in interlayers by static electricity, such as dust and a foreign matter, etc. poses a problem, and has the outstanding antistatic engine performance has been demanded.

[0004] However, for the interlayer which consists of the above-mentioned plasticization polyvinyl-acetal resin, surface electrical resistance is 10¹³ ohm/cm². It was extent, and in order to be hard to be charged generally and to use it, being fastened between glass plates, it was rare to be taken into consideration about the antistatic engine performance.

[0005] If the approach of the electrification prevention by the antistatic agent widely used for the transparent plastic on the other hand was applied to such an interlayer, since the adhesive property of an interlayer and glass, transparency, endurance, etc. would be spoiled, a suitable combination was not found out.

[0006] Although the interlayer for glass laminates which a non-ion system antistatic agent is scoured by JP,7-223849,A, or is applied to it as what solved the above-mentioned trouble was indicated, generally, the non-ion system antistatic agent required many contents, in order to obtain sufficient antistatic engine performance, and had the problem that possibility of having a bad influence on interlayer physical properties was large.

[0007] Moreover, although adipic-acid dihexyl, triethylene glycol di-2-ethyl butyrate, etc. are generally used for the interlayer as a plasticizer, there is a trouble that the outbreak of a fire at the time of an autoclave and the trim cut activity of the glass laminate edge behind an autoclave are difficult, in the case of glass laminate manufacture, and the plasticizer of a high-boiling point has been expected more at it.

[0008]

[Problem(s) to be Solved by the Invention] In order that this invention may solve the above-mentioned conventional trouble, it is excellent in the fundamental engine performance as interlayers for glass laminates, such as transparency, lightfastness, weatherability, shock resistance, and an adhesive property, and is excellent in moisture resistance and antistatic nature, and fear of the outbreak of a fire at the time of an autoclave reduces it further in the case of glass laminate manufacture, and it makes it a technical problem to offer the interlayer for glass laminates whose trim cut workability of a glass laminate edge improved.

[0009]

[Means for Solving the Problem] The interlayer for glass laminates by invention (henceforth invention 1) according to claim 1 The polyvinyl-acetal resin 100 weight section, TORIECHIRENGURIKORUJI 2-ethylhexanoate 20 - 60 weight sections, It is the interlayer for glass laminates which the mixture 0.01 - the 0.1 weight sections, and the carbon number of 2-ethyl butanoic acid magnesium and magnesium acetate become from the carboxylic-acid potassium salt 0.005 of 4-12 - the 0.5 weight sections. The above-mentioned mixture is characterized by being the mixture which fills the relation between $0.5 < \text{2-ethyl butanoic acid magnesium / magnesium acetate} < 3.0$ with a weight ratio.

[0010] In the interlayer for glass laminates by the above-mentioned invention 1, as for the interlayer for glass laminates by invention (henceforth invention 2) according to claim 2, polyvinyl-acetal resin is characterized by whenever [butyral-ized] being 66-72-mol the polyvinyl butyral resin it is [polyvinyl butyral resin] %.

[0011] It is characterized by for the glass laminate by invention (henceforth invention 3) according to claim 3 making invention 1 or the interlayer for glass laminates by 2 intervene between the glass of a pair, and making it come at least to unify it. Hereafter, this invention is explained to a detail.

[0012] As polyvinyl-acetal resin used in the interlayer by invention 1, the polyvinyl butyral resin (henceforth PVB) whenever [butyral-ized / whose] is 66-72-mol % is used suitably. PVB whenever [butyral-ized / whose] is 67.5-70-mol % is more desirable. in order that the absorptivity of the interlayer from which whenever [above-mentioned butyral-ized] is obtained less than [66 mol %] may go up -- the glass laminate periphery section -- albinism -- a lifting -- easy -- when it may become and whenever [butyral-ized] exceeds 72-mol % conversely, the mechanical strength of the interlayer obtained may become inadequate

[0013] It is not limited especially as the manufacture approach of Above PVB. For example, polyvinyl alcohol Dissolve in hot water (it is hereafter called PVA), and the obtained water solution is held at 10-20 degrees C. A necessary butyraldehyde and a necessary acid catalyst are added, an acetalization reaction is advanced, and after carrying out the temperature up of the temperature, holding it subsequently to 70 degrees C and completing a reaction, the method of performing neutralization, rinsing, and desiccation and obtaining PVB powder etc. is mentioned.

[0014] As the above PVA, the thing of average degree of polymerization 1000-2500 is desirable. Manufacture of an interlayer may become difficult, when the penetration-proof of the glass laminate with which this average degree of polymerization is obtained less than by 1000 etc. may fall and average degree of polymerization exceeds 2500.

[0015] Moreover, Above PVA has that desirable whose saponification degree is more

than 95 mol %. When a saponification degree is less than [95 mol %], the transparency of the interlayer obtained, thermal resistance, lightfastness, weatherability, etc. may become inadequate.

[0016] In the interlayer by invention 1, it is required as a plasticizer to carry out 20-60 weight section content of the TORIECHIRENGURIKORUJI 2-ethylhexanoate (henceforth 3GO). If the penetration-proof of the glass laminate with which the content of 3GO(s) is obtained under in 20 weight sections etc. falls and the content of 3GO exceeds 60 weight sections, it will become easy to carry out bleed out of the 3GO(s) from the interlayer obtained, and the transparency and the adhesive property of an interlayer will fall.

[0017] The above-mentioned 3GO can be manufactured by making triethylene glycol and 2-ethylhexanoic acid more than the 2 double equivalent react under existence of a catalyst.

[0018] In the interlayer by invention 1, it is required to carry out 0.01-0.1 weight section content of the mixture of 2-ethyl butanoic acid magnesium (henceforth C6 Mg) and magnesium acetate (henceforth C2 Mg), and it is the 0.03 - 0.08 weight section preferably. The transparency of the glass laminate obtained as since the adhesive strength adjustment effectiveness is lost by the content of the mixture of above-mentioned C6 Mg and C2 Mg less than by 0.01, if penetration-proof falls and the 0.1 weight section is exceeded conversely and moisture resistance get worse.

[0019] Moreover, in the interlayer by invention 1, it is required to be the mixture with which the above-mentioned mixture fills the relation of $0.5 < C_6 \text{ Mg} / C_2 \text{ Mg} < 3.0$ with a weight ratio, and it is $1.0 < C_6 \text{ Mg} / C_2 \text{ Mg} < 2.5$ in a weight ratio preferably. Less than by 0.5, above-mentioned $C_6 \text{ Mg} / C_2 \text{ Mg}$ (weight ratio) will become unstable [the penetration-proof of the glass laminate obtained], if the moisture resistance of the glass laminate with which the above-mentioned mixture causes condensation in an interlayer, and is obtained falls and 2.5 is exceeded conversely.

[0020] Above-mentioned C6 Mg or C2 Mg can be manufactured by making 2-ethyl butanoic acid or an acetic acid, and a magnesium oxide react under existence of a catalyst, respectively.

[0021] These magnesium salt can make good penetration-proof of the glass laminate which has the function to make the adhesive strength between an interlayer and glass hold in the proper range, consequently is obtained by existing in the form of a salt, respectively, without ionizing in an interlayer, and drawing a water molecule near.

[0022] Moreover, since these magnesium salt is distributed over an interlayer front face by high concentration by being used together, without existing as complex in an interlayer and condensing, it demonstrates the adhesive strength adjustment effectiveness excellent in the little addition. Consequently, the glass laminate obtained becomes what has the outstanding penetration-proof, and the albinism by moisture absorption hardly raises it, either.

[0023] In the interlayer by invention 1, it is required for the carboxylic-acid potassium salt of 4-12 to contain [the carbon number]. Less than by four, the carbon number of the above-mentioned carboxylic acid causes aging of the adhesive strength of the interlayer obtained, in order that carboxylic-acid potassium salt may segregate too much on an interlayer front face, if the electrification nature prevention effectiveness of the interlayer obtained falls and a carbon number exceeds 12 conversely, since condensation of a

carboxylic-acid potassium salt particle takes place in an interlayer.

[0024] Moreover, in the interlayer by invention 1, it is required to carry out 0.005-0.5 weight section content of the carboxylic-acid potassium salt of 4-12 for the above-mentioned carbon number. If the electrification nature prevention effectiveness of an interlayer that the content of carboxylic-acid potassium salt is obtained under in the 0.005 weight section falls and the 0.5 weight section is exceeded conversely, the moisture resistance of the interlayer obtained will fall.

[0025] The carboxylic-acid potassium salt of 4-12 makes the charge of static electricity distribute and conduct in an interlayer, and the above-mentioned carbon number has the effectiveness of preventing the electrification nature of the interlayer obtained.

[0026] Moreover, the above-mentioned carbon number is not limited especially as carboxylic-acid potassium salt of 4-12, for example, a butanoic acid potassium, an isobutyl acid potassium, 2-ethyl butanoic acid potassium, an octanoic-acid potassium, a decanoic-acid potassium, a potassium oxalate, a malonic-acid potassium, the potassium succinate, a glutaric-acid potassium, an adipic-acid potassium, etc. are mentioned. These may be used independently and may be used together two or more sorts.

[0027] Furthermore, various additives, such as a stabilizer for preventing degradation of an interlayer, an antioxidant, and an ultraviolet ray absorbent, can be used for the interlayer of invention 1 if needed, and these may be used independently and may be used together two or more sorts.

[0028] Especially as a stabilizer, it is not limited, for example, a hindered amine system stabilizer like trade name ADEKA stub LA-57 by Asahi Denka Kogyo K.K. etc. is mentioned, and these may be used independently and may be used together two or more sorts.

[0029] Especially as an anti-oxidant, it is not limited, for example, phenolic antioxidants, such as t-butylhydroxytoluene (for example, Sumitomo Chemical Co., Ltd. make, trade name Sumi Reiser BHT) and tetrakis-[methylene-3-(3'-5'-JI t-butyl -4'-hydroxyphenyl) propionate] methane (for example, the Ciba-Geigy make, trade name IRUGA NOX 1010), etc. are mentioned, and these may be used independently and may be used together two or more sorts.

[0030] It is not limited especially as an ultraviolet ray absorbent. For example, 2-(2'-hydroxy-5'-methylphenyl) benzotriazol For example, (the Ciba-Geigy make and trade name tinuvin P), 2-(2' - hydroxy-3', 5'-JI t-butylphenyl) benzotriazol (Ciba-Geigy make and trade name tinuvin 320) 2-(2'-hydroxy-3'-t-butyl -5'-methylphenyl)-5-chlorobenzotriazole [for example,] For example, (the Ciba-Geigy make and trade name tinuvin 326), 2-(2' - hydroxy-3', 5'-JIAMIRU phenyl) benzotriazol (For example, the Ciba-Geigy make, trade name tinuvin 328) etc. -- a benzotriazol system ultraviolet ray absorbent, the Asahi Denka Kogyo K.K. make, a hindered amine system ultraviolet ray absorbent like trade name ADEKA stub LA-57, etc. are mentioned, and these may be used independently and may be used together two or more sorts.

[0031] Especially the manufacture approach of the interlayer by invention 1 is not limited. 3GO(s) of the specified quantity, the mixture of C6 Mg and C2 Mg, and a carbon number to Above PVB The carboxylic-acid potassium salt of 4-12, One sort of various additives or two sorts or more can be blended if needed, and it can obtain as resin film by fabricating the kneading object which supplied this compound to the roll mill, kneaded, and was obtained in the shape of a sheet with a press-forming machine, a calendering

roll, an extruder, etc.

[0032] Next, it is characterized by for the glass laminate by invention 3 making invention 1 or the interlayer for glass laminates by 2 intervene between the glass of a pair, and making it come at least to unify it.

[0033] Especially as the above-mentioned glass, it is not limited, for example, organic glass, such as inorganic glass, such as float plate glass, polished plate glass, figured glass, heat ray absorption plate glass, and coloring sheet glass, or a polycarbonate plate, and a polymethylmethacrylate plate, is mentioned.

[0034] Although the approach generally used is employable as the manufacture approach of the glass laminate by invention 3 For example, invention 1 or the interlayer of 2 is fastened with the float glass of two sheets. It holds for 30 minutes at 90 degrees C within oven, putting this fastening object into the rubber back, and making it a vacuum. After taking out this fastening object from the rubber back, a transparent glass laminate can be obtained by carrying out a hot press and making it unify at the pressure of 13kg/cm², and the temperature of 140 degrees C within an autoclave.

[0035]

[Embodiment of the Invention] Although an example is given to below in order to explain this invention in more detail, this invention is not limited only to these examples. In addition, the "section" in an example means the "weight section."

[0036] (Example 1)

(1) It heated and dissolved in 95 degrees C, supplying and agitating the saponification degree PVA[% of] of 99.2 mols198 section (4.5 mol considerable amount of vinyl alcohol) to a reactor with churning equipment by the synthetic ion-exchange-water 2900 section of PVB, and average degree of polymerization 1700. After PVB deposited having cooled this solution at 30 degrees C, having added the 35-% of the weight hydrochloric-acid 208 section (2.1 mols) and the n-butylaldehyde 152 section (2.1 mols), having lowered solution temperature subsequently to 2 degrees C, and holding this temperature, the temperature up of the solution temperature was carried out to 30 degrees C, and it was held for 5 hours. Then, the sodium-hydrogencarbonate 156 section (1.8 mols) was added, it neutralized, rinsing and desiccation were performed, and 69-mol % PVB powder was obtained whenever [butyral-ized].

[0037] 3GO(s) as the PVB100 section obtained by manufacture (1) of an interlayer, and a plasticizer (2) The 39 sections, The mixture [of the C6 Mg0.02 section and the C2 Mg0.01 section] [C6 Mg/C2 Mg(weight ratio) =2.0] and 2-ethyl butanoic acid potassium 0.02 section is supplied to a roll mill. It is the kneading object kneaded and obtained with a press-forming machine 150 degrees C and 100kg/cm² Press forming was carried out for 30 minutes under conditions, and the interlayer with a thickness of 0.8mm was obtained.

[0038] (3) After having fastened the interlayer obtained by manufacture (2) of a glass laminate between 30cmx30cm and the float glass with a thickness of 2.5mm of two sheets, putting this fastening object into the rubber back and holding for 20 minutes by degree of vacuum 20torr, it put in in oven in the condition [having made it the vacuum], and held for 30 minutes at 90 degrees C. The hot press of the fastening object taken out from the rubber back was carried out under conditions with a pressure [of 13kg/cm²], and a temperature of 150 degrees C within the autoclave, and the glass laminate was obtained.

[0039] (4) The following approaches estimated the engine performance (3. the volatility

of a plasticizer, 4. surface electrical resistance) of the interlayer obtained by the engine performance (1. a pan mel value, 2. moisture resistance) of the glass laminate obtained by evaluation (3), and (2). The result was as being shown in Table 2.

[0040] 1. The hammer whose head is 0.45kg struck the glass laminate which left to with a pan mel value [of -18**0.6 degrees C] temperature for 16 hours, and carried out temperature control to it, and it ground until the particle diameter of glass was set to 6mm or less. Subsequently, it judged by sample [limit] which carried out grade attachment of whenever [exposure / of the interlayer after glass carries out partial avulsion] beforehand, and the result was expressed as a pan mel value in accordance with the criterion shown in the following table 1. In addition, the pan mel value was measured about two conditions after leaving the first stage and a glass laminate for four weeks at 50 degrees C. The adhesive strength of an interlayer and glass is so large that the above-mentioned panmel value is large, and the adhesive strength of an interlayer and glass is so small that a pan mel value is small.

[0041]

[Table 1]

[判定基準]

中間膜の露出度(面積%)	パンメル値
100	0
90	1
85	2
60	3
40	4
20	5
10	6
5	7
2以下	8

[0042] 2. Immediately after leaving the damp-proof glass laminate for two weeks under 80 degrees C and the ambient atmosphere of 95% of relative humidity, it took out and the milkiness distance (mm) from a glass laminate periphery edge was measured.

[0043] 3. The initial mass (A) of the interlayer of an volatile fixed area of a plasticizer was measured. Subsequently, after leaving this interlayer in 150-degree C oven for 1 hour, it took out, heating Shigekazu Ushiro (B) was measured, and the heating loss (% of the weight) of an interlayer was computed by the bottom type. The volatility of a plasticizer is so low that the above-mentioned heating loss is small, and the volatility of a plasticizer is so high that heating loss is large.

Heating loss (% of the weight) = $(A-B/A) \times 100$ [0044] 4. after drying a surface-electrical-resistance profit **** interlayer in a 24-hour desiccator, surface electrical resistance was measured with the surface-electrical-resistance measuring device (the Toa Electronics, Ltd. make, DSM-8103). Surface electrical resistance made good under 1.0×10^{13} ohms / **, and made more than it the defect.

[0045] (Example 2) In manufacture of an interlayer, as shown in Table 2, the content of C6 Mg was made into the 0.025 sections, and the interlayer and the glass laminate were obtained like the example 1 except having used the hexyl acid potassium 0.01 section instead of the 2-ethyl butanoic acid potassium 0.02 section. C6 Mg/C2 Mg in the above-

mentioned interlayer (weight ratio) was 2.5.

[0046] (Example 3) In manufacture of an interlayer, as shown in Table 2, the content of C₂ Mg was made into the 0.02 sections, and the interlayer and the glass laminate were obtained like the example 1 except having used the octanoic-acid potassium 0.2 section instead of the 2-ethyl butanoic acid potassium 0.02 section. C₆ Mg/C₂ Mg in the above-mentioned interlayer (weight ratio) was 1.0.

[0047] (Example 1 of a comparison) In manufacture of an interlayer, as shown in Table 2, the interlayer and the glass laminate were obtained like the example 1 as a plasticizer except having used the adipic-acid dihexyl 39 section instead of the 3GO39 section.

[0048] (Example 2 of a comparison) In manufacture of an interlayer, as shown in Table 2, the interlayer and the glass laminate were obtained like the example 1 except having made the content of C₆ Mg into the 0.04 sections. C₆ Mg/C₂ Mg in the above-mentioned interlayer (weight ratio) was 4.0.

[0049] (Example 3 of a comparison) In manufacture of an interlayer, as shown in Table 2, the interlayer and the glass laminate were obtained like the example 1 except having used the propionic-acid potassium 0.05 section instead of the 2-ethyl butanoic acid potassium 0.02 section.

[0050] (Example 4 of a comparison) In manufacture of an interlayer, as shown in Table 2, the interlayer and the glass laminate were obtained like the example 1 except having used the oleic acid potassium 0.05 section instead of the 2-ethyl butanoic acid potassium 0.02 section.

[0051] The engine performance of six kinds of glass laminates obtained in examples 2 and 3 and the examples 1-4 of a comparison and an interlayer was evaluated like the case of an example 1. The result was as being shown in Table 3.

[0052]
Table 2

試験 番号	樹脂 アクリル化度 (mol%)	可塑剤 (重量部)	マグネシウム塩				種類	カリウム塩	添加剤数 (重量部)
			C6Mg (重量部)	C2Mg (重量部)	比率 $\text{C6Mg}/\text{C2Mg}$	添加部数 (重量部)			
実験1	6.9	3GO	0.02	0.01	2.0	0.03	2-エチル醋酸カリム	0.02	
実験2	6.9	3GO	0.025	0.01	2.5	0.035	ヘキシル酢酸カリム	0.01	
実験3	6.9	3GO	0.02	0.02	1.0	0.04	オクタン酸カリム	0.2	
実験4	6.9	3GO	0.02	0.01	2.0	0.03	2-エチル醋酸カリム	0.02	
実験5	6.9	3GO	0.04	0.01	4.0	0.05	2-エチル醋酸カリム	0.02	
比較例4	6.9	3GO	0.02	0.01	2.0	0.03	プロピオン酸カリム	0.05	
		3G	0.02	0.01	2.0	0.03	オレイン酸カリム	0.05	

[0053]

[Table 3]

		パンメル値		白化距離 (mm)	加熱減量 (重量%)	表面抵抗 (10^{13} Ω/□)
		初期	経時			
実施例	1	4	4	1. 5	1	0. 2
	2	5	5	2	1	0. 3
	3	4	3. 5	1. 5	2	0. 1
比較例	1	4	4	2	8	0. 2
	2	4	1. 5	2	1	0. 2
	3	4	4	2	1	4. 0
	4	4	1	2	1	0. 5

[0054] The glass laminate of the example by this invention had a proper pan mel value also in any of first stage and passage-of-time Ushiro, its adhesive property was stable, and moisture resistance was also excellent so that clearly from Table 3. Furthermore, the interlayer of the example by this invention had small heating loss, and there was little volatilization of a plasticizer and it was excellent also in antistatic nature.

[0055] On the other hand, the interlayer of the example 1 of a comparison using adipic-acid dihexyl as a plasticizer had large heating loss, and there was much volatilization of a plasticizer. Moreover, the pan mel value of Ushiro with the passage of time fell, and the glass laminate using the interlayer of the example 2 of a comparison for which C6 Mg/C2 Mg (weight ratio) exceeds 3.0 had the unstable adhesive property.

[0056] Moreover, the interlayer of the example 3 of the comparison using less than four carboxylic-acid potassium salt of a carbon number has high surface electrical resistance, and is inferior to antistatic nature in it. Furthermore, the pan mel value of Ushiro with the passage of time fell, and the glass laminate using the interlayer of the example 4 of a comparison using the carboxylic-acid potassium salt to which a carbon number exceeds 12 had the unstable adhesive property.

[0057]

[Effect of the Invention] As stated above, the interlayer of this invention satisfies the fundamental engine performance as interlayers for glass laminates, such as transparency, lightfastness, weatherability, shock resistance, and penetration-proof, and an adhesive property is stabilized, it is excellent also in moisture resistance, and there is little volatility of a plasticizer and it is excellent also in antistatic nature. Therefore, the interlayer and glass laminate of this invention are suitably used as objects for cars, such as an automobile, a windowpane of ***** etc.

[Translation done.]